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ABSTRACT

Randomly oriented polycrystalline copper of 99.995% purity was tested in tension at temperatures of 626, 496 and 406°C. The grain size range investigated was from 0.03 mm to 0.7 mm. Grain sizes were produced by two techniques: (1) varying the amount of prior cold work and the annealing time at constant annealing temperature so as to obtain various recrystallized grain sizes with minimum grain growth and (2) holding the prior cold work constant while varying the annealing time or temperature so as to obtain varying grain sizes by grain growth. Polycrystalline samples (grain size of 0.03 mm) with a strong [001] (100) texture were also studied.

The relationship between the steady state creep rate, $\dot{\epsilon}_s$, and grain size was found to be the same independent of the technique used to prepare the various grain sizes. For specimens with grain diameters above about 0.1 mm, $\dot{\epsilon}_s$ appeared to be independent of grain size while for smaller grain diameters, $\dot{\epsilon}_s$ increased slightly with decreasing grain size. Strongly textured polycrystalline copper, which contained low angle grain boundaries, exhibited steady state creep rates that were slightly lower than those observed in randomly oriented copper of the same grain size. The results are explained by considering the contribution of grain boundary shearing to the total strain and the effect of grain size on the resulting creep substructure.

INTRODUCTION

Experimental observations concerning the influence of grain size on steady state creep rates are varied and oftentimes conflicting. Numerous early investigators found that an increase in the grain size resulted in a decrease in the high temperature steady state creep rate, $\dot{\epsilon}_s$ [1-7]. Other workers have found that $\dot{\epsilon}_s$ decreases with increasing grain size up to some optimum grain size and then increases with a further increase in grain size[8-11]. It has been suggested that $\dot{\epsilon}_s$ decreases with an increase in grain size only because the range of grain sizes studied normally lies to the left of the grain size for optimum creep resistance. However, Sherby[12] and Feltham and his co-workers[13, 14] have found that even for small grain sizes $\dot{\epsilon}_s$ does not always decrease with an increase in grain diameter but may be proportional to the square of the grain diameter.

A possible explanation for the different creep rate - grain size relationships which have been observed is that in most experimental studies different grain sizes have been prepared by annealing at various temperatures. For metals in which the impurity concentration is high (greater than about 0.1 atomic percent) this type of treatment may lead to different impurity distributions which may subsequently influence the observed creep characteristics. In addition, Parker[15] has presented data which indicate that the observed creep rate - grain size relationship may depend on the type of annealing treatment used to obtain the different grain sizes, even in the case of pure metals. Parker proposes that the production of different grain sizes by conventional techniques (i.e., cold working and then annealing at successively higher temperatures) results not only in a change in grain size but also in a change in the nature of the boundaries, the larger angle high-energy boundaries tending to disappear preferentially

during grain growth. Thus, although the metal contains a random distribution of high and low energy boundaries immediately upon primary recrystallization, subsequent grain growth alters this distribution to a preponderance of low energy boundaries. On this basis, Parker concludes that when different grain sizes are produced by the grain growth method, the steady state creep rate should decrease with increasing grain size. This is because the large grain size samples would have primarily low-angle boundaries which would be expected to be poor sources of vacancies, leading to a low dislocation climb rate. On the other hand, if different grain sizes are prepared with a minimum of grain growth, leading to a random distribution of grain boundary misorientations, then Parker concludes that the steady state creep rate should increase with increasing grain size since there are less boundary barriers to dislocations with increasing grain size. Parker considers that grain boundary shearing is not an important factor in high temperature creep.

The present investigation was initiated in order to elucidate further the influence of grain size on the creep resistance of pure metals. Special emphasis was placed on the possible role of annealing treatment, grain boundary misorientation, grain boundary shearing and substructure on the steady state creep rate.

MATERIAL AND EXPERIMENTAL PROCEDURE

Copper was chosen as the test material because of its availability in reasonably high purity and because extensive data exist regarding the influence of various annealing treatments on the average misorientation of the grain boundaries. Grain sizes were prepared by three different methods:

(1) The variable strain technique. Material was deformed various

amounts by cold rolling and then annealed at a specific temperature until primary recrystallization was complete. Grain sizes prepared by this technique were obtained with a minimum of grain growth.

(2) The variable temperature technique. Material was deformed a specific amount by cold rolling and then annealed for a specific time at various temperatures. This method resulted in a variety of grain sizes produced primarily by grain growth.

(3) The variable annealing time technique. Material was deformed a specific amount by cold rolling and then annealed at a specific temperature for various lengths of time. As in method (2) grain sizes prepared by this technique were achieved primarily by grain growth.

The creep characteristics of these samples were studied at various stresses and temperatures. Measurements were made regarding the influence of grain size on both the dislocation substructure and the contribution of grain boundary sliding to the total strain. In addition, the creep data from these studies were compared with data from strongly cube textured copper to study the influence of grain boundary misorientation on creep.

The material used in this investigation was high purity OFHC copper. Spectrographic and chemical analyses showed only 0.001% Ni, 0.001% Al, <0.001% Mg and 0.002% O_2 as impurities, indicating a purity of about 99.995%. All material was first reduced 50% by cold rolling and then recrystallized at 700°C. This procedure has been shown[16] to remove any prior texture and to produce a randomly oriented polycrystalline structure. Following this randomizing treatment, the copper was cold rolled the desired amount

and then given the final anneal. Grain size determinations were made by counting the number of intersections, M, of grain boundaries with randomly oriented lines of total length, L, and setting the average grain diameter equal to L/M. For most determinations over two hundred grain boundaries were counted. Twin boundaries were not counted as boundaries in the determination of grain size.

The pertinent data regarding grain sizes produced by the variable strain technique are given in Table 1. The data concerning the different grain sizes produced by the variable annealing temperature technique are listed in Table 2. Two sets of samples prepared by this technique were given a secondary low temperature treatment following the primary anneal. The purpose of these low temperature annealing treatments was to eliminate differences in impurity distributions arising from the different temperature anneals. Grain sizes prepared by variable annealing times at a constant annealing temperature are listed in Table 3.

Low angle grain boundary specimens were prepared with a strong [001] (100) texture by cold rolling 97% and then annealing in a vacuum furnace at 700°C for 15 minutes. The average grain diameter for the cube textured material was 0.03 mm. The average grain boundary angle was determined by measuring slip traces in adjacent grains and was found to be about 5°.

All creep specimens were flat sheet tensile specimens. They were machined following the final cold rolling treatment and polished with 3/0 emery paper prior to annealing. The thickness of the gauge section was such as to always maintain at least 25 grains in the specimen cross-section. Constant stress creep tests were performed using Andrade-Chalmers type lever arms. All creep tests were conducted in a dry deoxidized hydrogen atmosphere. Temperature measurement was made by means of chromel-alumel

TABLE 1 - Annealing treatments and constant stress creep data for grain sizes obtained by variable strain technique. All annealing done in hydrogen unless otherwise noted.

Spec. No.	Final Reduction %	Anneal Temp., °C	Anneal Time, min.	Test Temp., °C	Stress, 10^8 dynes/cm^2	Grain Diameter, mm.	$\dot{\epsilon}_s$ 10^{-5} min^{-1}
28*	50	700	5	496	2.07	0.03	4.2
34	35	700	7	496	2.07	0.074	2.6
200	27	700	10	496	2.07	0.11	2.05
203	27	700	10	496	2.07	0.11	1.85
205	27	600	20	406	3.45	0.085	2.72
206	6	700	60	496	2.07	0.30	1.97
209	6	800	30	626	2.07	0.46	23.6
210	10	600	60	406	3.45	0.24	1.29
211	10	700	40	406	3.45	0.22	1.53
212	10	600	60	406	3.45	0.24	1.56
213	10	700	40	496	2.07	0.22	2.35
215	16	700	20	496	2.07	0.19	1.76
218	16	800	20	626	2.07	0.37	26.8
221	21	700	15	496	2.07	0.15	2.1
222	21	600	25	406	3.45	0.12	1.7
223	21	800	12	626	2.07	0.31	30.5
225	24	800	12	626	2.07	0.26	26.7
227	24	600	20	406	3.45	0.095	1.8
228	24	700	15	496	2.07	0.14	2.3
230	30	700	10	496	2.07	0.093	2.14
231	30	700	10	496	2.07	0.093	2.5
232**	30	800	8	626	2.07	0.22	56.0
233	30	600	15	406	3.45	0.069	1.82
234	30	700	10	406	3.45	0.093	1.5
237**	36	800	5	626	2.07	0.16	50.6
241	41	700	5	496	2.07	0.053	3.04
242**	41	800	5	626	2.07	0.11	100
243	41	700	5	496	2.07	0.053	3.35
245	46	700	3	496	2.07	0.046	2.71
248	46	600	6	406	3.45	0.046	2.9
250	49	600	6	406	3.45	0.033	3.3
252	49	700	3	496	2.07	0.040	3.62
253	49	700	3	496	2.07	0.040	3.8
254	49	700	3	406	3.45	0.040	2.32
255	49	600	6	406	3.45	0.033	3.3

* Annealed in vacuum

** Recrystallized during test

TABLE 2 - Annealing treatments and constant stress creep data for grain sizes obtained by variable annealing time technique. All annealing done in hydrogen.

Spec. No.	Final Reduction %	Anneal Temp., °C	Anneal Time, min.	Test Temp., °C	Stress, 10^8 dynes/cm^2	Grain Diameter, mm.	$\dot{\epsilon}_s$ 10^{-5} min^{-1}
225	50	700	3200	496	2.07	0.48	2.5
250	49	600	6	406	3.45	0.033	3.3
252	49	700	3	496	2.07	0.04	3.62
253	49	700	3	496	2.07	0.04	3.8
255	49	600	6	406	3.45	0.033	3.3
260	50	600	10	406	3.45	0.066	2.43
261	50	600	50	406	3.45	0.11	2.18
262	50	600	500	406	3.45	0.15	1.82
263	50	600	1500	406	2.07	0.24	1.84
320	50	700	50	496	2.07	0.25	2.92
321	50	700	500	496	2.07	0.40	2.7
322	50	700	25	496	2.07	0.14	2.58
324	50	700	3200	496	2.07	0.48	2.32

TABLE 3 - Annealing treatments and constant stress creep data for grain sizes obtained by variable temperature technique. All annealing done in hydrogen unless otherwise noted.

Spec. No.	Final Reduction %	Anneal Temp., °C	Anneal Time, min.	Test Temp., °C	Stress 10^8 dynes/cm^2	Grain Diameter, mm.	$\dot{\epsilon}_s$ 10^{-5} min^{-1}
300	50	700	5	496	2.07	0.063	2.1
302	50	850	5	496	2.07	0.23	4.22
303	50	900	5	496	2.07	0.41	3.96
304	50	975	5	496	2.07	0.51	4.55
306	50	1050	5	496	2.07	0.71	4.5
307+	50	700	5	496	2.07	0.11	2.4
308	50	775	5	496	2.07	0.17	3.72
309+	50	850	5	496	2.07	0.23	3.44
310+	50	900	5	496	2.07	0.41	3.3
311+	50	975	5	496	2.07	0.51	3.2
312+	50	1050	5	496	2.07	0.71	4.6
326*	50	1050	10	496	2.07	0.77	2.08
330**	50	1050	5	496	2.07	0.71	2.6
331++	50	1050	5	496	2.07	0.71	2.52
332++	50	850	5	496	2.07	0.23	2.28
333++	50	975	5	496	2.07	0.51	2.6
334*	50	850	10	496	2.07	0.19	2.32
335*	50	975	10	496	2.07	0.48	2.25

\+ given 360 minute annealing treatment at 600°C prior to testing
* annealed in vacuum
** given 1600 minute annealing treatment at 700°C prior to testing
++ given 800 minute annealing treatment at 700°C prior to testing

thermocouples in direct contact with the specimen. The specimen thermocouples were periodically checked against new thermocouples to insure accuracy. Temperature control was normally better than $\pm$ 1°C during the course of any test. The creep strain was measured using a linear variable differential transformer and it was possible to accurately measure incremental displacements of 2×10^{-3} mm. The creep specimens' reduced gage sections were 44.5 mm long and 6.4 mm wide.

The technique employed for determining the amount of grain boundary shearing was that developed by McLean[17]. A series of grid lines was scribed on the surface of the sample and the average value of the longitudinal displacement at a grain boundary was obtained by averaging 100 separate displacements.

Samples intended for transmission electron microscopy studies were tested on a creep unit equipped with a sliding furnace. Following testing the entire furnace was lowered and the specimen was cooled rapidly under load. The cooling rate above 100°C was approximately 300°C per minute. Specimens were maintained in a hydrogen atmosphere during the entire cooling cycle. It was hoped that the rapid cool while under load would preserve the existing creep substructure. After testing, samples were chemically polished to a thickness of $\sim$ 0.05 mm using a solution of 50 ml HNO_3, 25 ml H_3PO_4 and 25 ml glacial acetic acid. Thin foils were then prepared by electropolishing in a solution of 67 ml methyl alcohol and 33 ml HNO_3 maintained at -30°C. All transmission microscopy was performed with a Hitachi Hu 11 electron microscope operating at 100 kv.

RESULTS

The majority of creep tests run on high purity copper yielded well defined regions of primary and steady state creep. A typical creep curve obtained at a stress of 3.45×10^8 dynes/cm^2 and a temperature of 406°C is shown in Figure 1. Under certain test conditions recrystallization occurred during testing and a true steady state creep rate was not obtained. For these tests a minimum creep rate, $\dot{\epsilon}_m$, is reported instead of a steady state rate. A creep curve exhibiting recrystallization during testing is shown in Figure 2.

Tables 1 - 3 list the observed steady state or minimum creep rates for the various samples tested.

A. INFLUENCE OF GRAIN SIZE AND ANNEALING TREATMENT ON $\dot{\epsilon}_s$

All of the steady-state creep rate - grain size data obtained, with the exception of some of the creep data obtained by the variable annealing temperature technique, obey the general relationship illustrated schematically in Fig. 3. This was found to be true regardless of annealing treatment or test conditions. The data which did not obey this relationship were from samples which were annealed at various high temperatures in hydrogen. It was demonstrated, however, that if samples were annealed in vacuum or given low temperature aging treatments prior to testing they behaved in accord with Fig. 3. The detailed results obtained from the various thermal-mechanical treatments are described in the following subsections.

1. Variable Strain Technique

The variation of $\dot{\epsilon}_s$ with grain size for samples prepared by the variable strain technique is given in Figures 4 - 6. The data in these

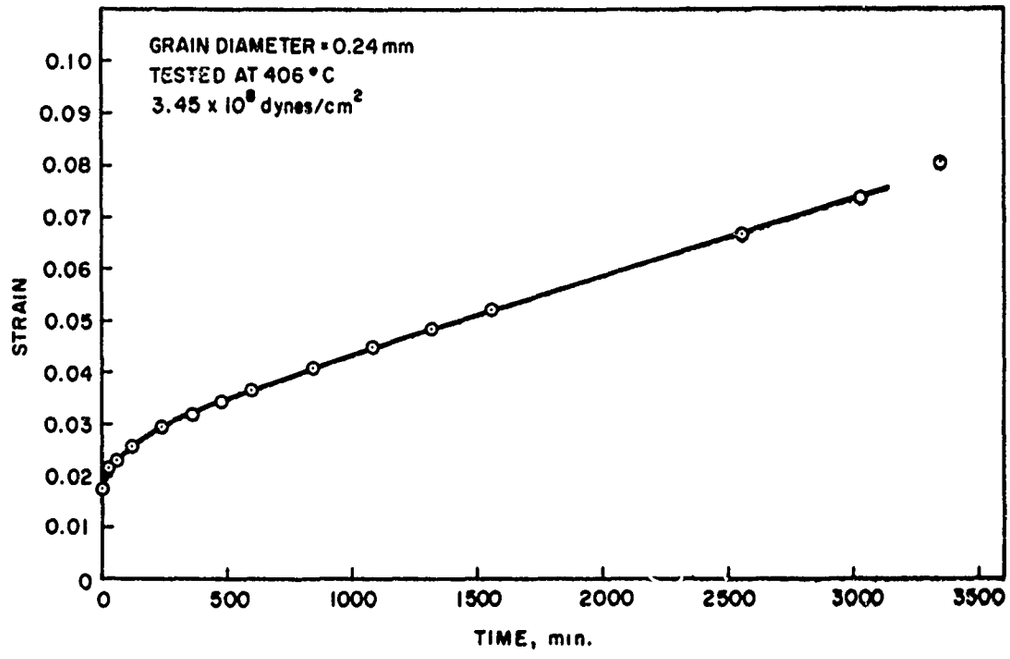

Fig. 1. Typical creep curve for high purity copper tested at 406°C and 3.45×10^8 dynes/cm^2.

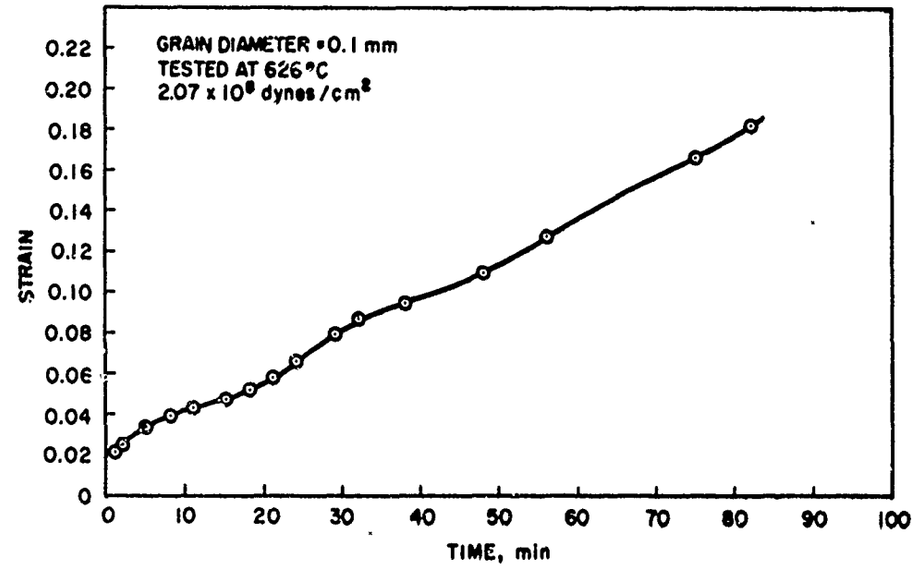

Fig. 2. Creep curve for high purity copper tested at 626°C and 2.07×10^8 dynes/cm^2 showing recrystallization during creep.

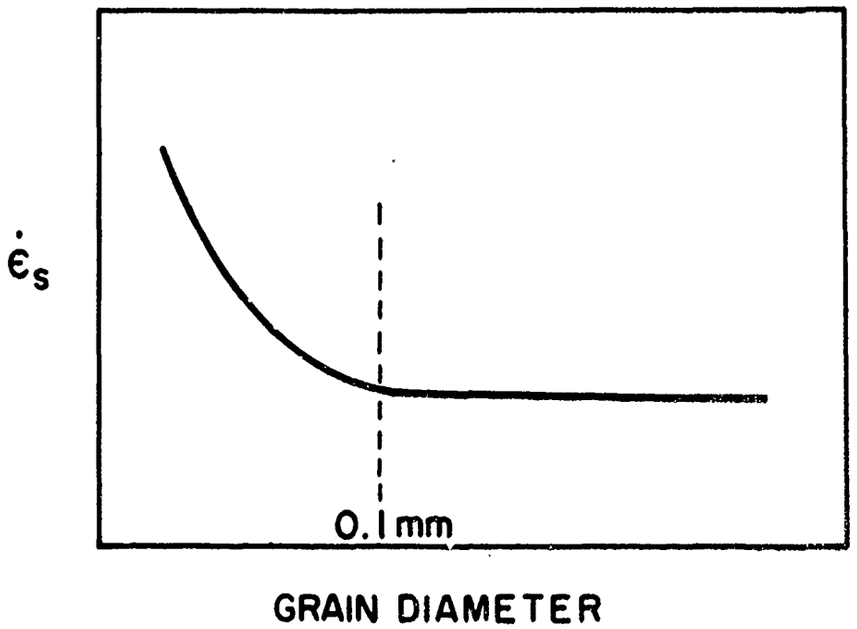

Fig. 3. Schematic representation of the general steady state creep rate - grain diameter relationship for high purity copper.

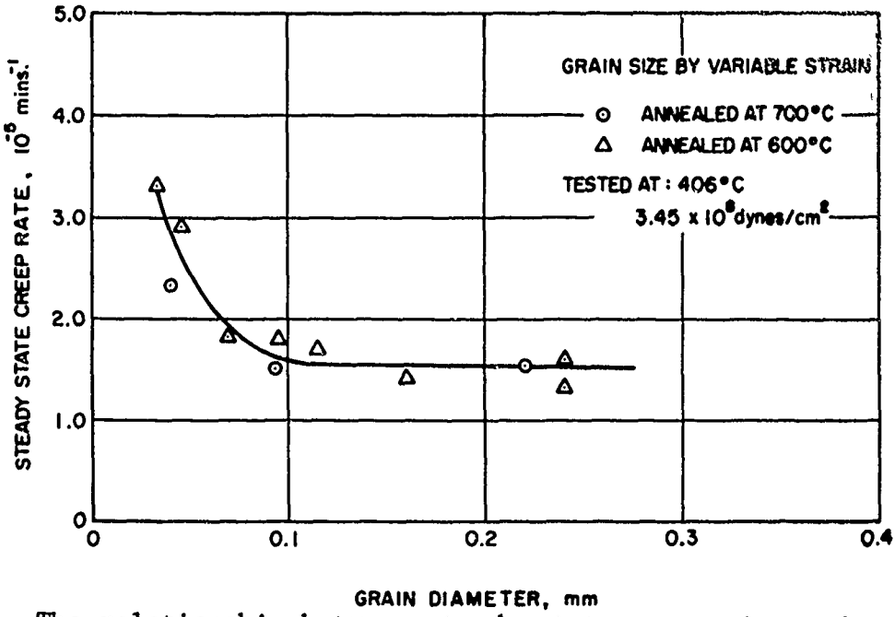

Fig. 4. The relationship between steady state creep rate and grain diameter for high purity copper tested at 406°C and 3.45 x 10^8 dynes/cm^2. Grain sizes produced by variable strain technique.

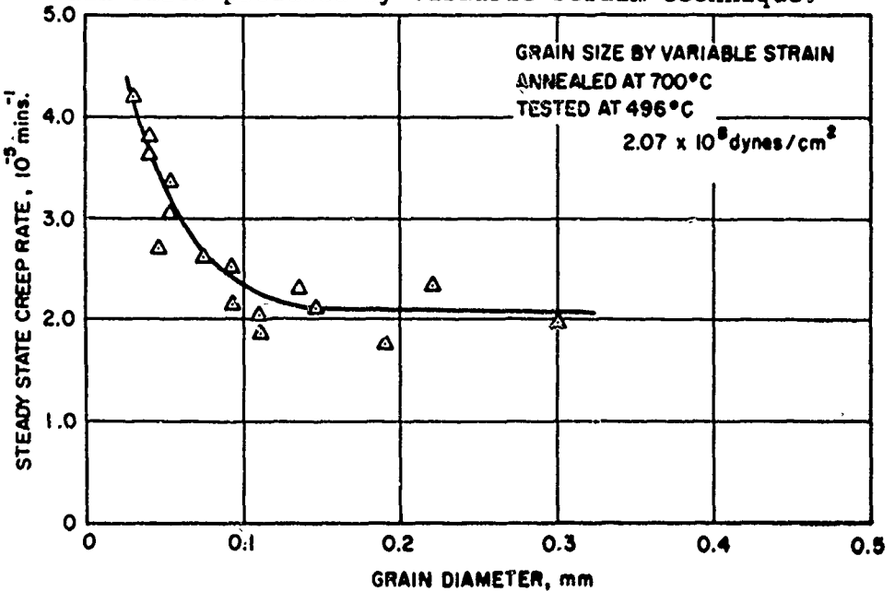

Fig. 5. The relationship between steady state creep rate and grain diameter for high purity copper tested at 496°C and 2.07 x 10^8 dynes/cm^2. Grain sizes produced by variable strain technique. Specimens annealed at 700°C.

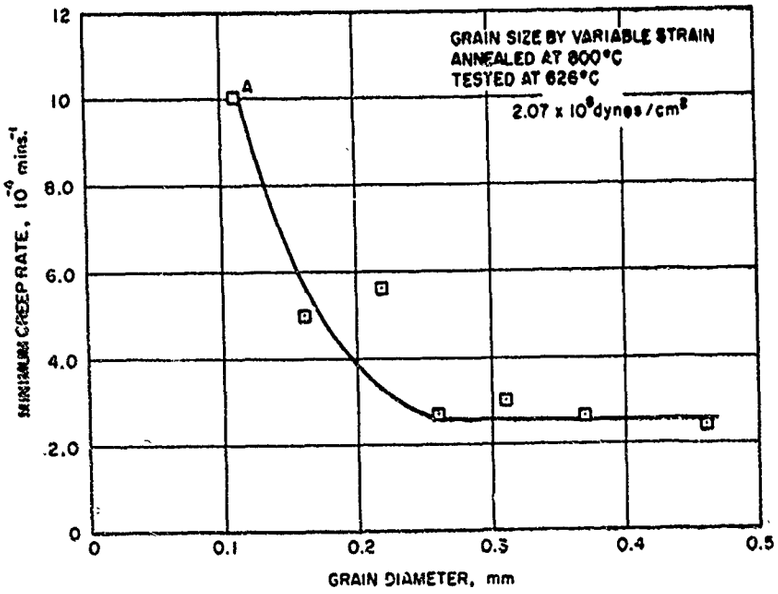

Fig. 6. The relationship between minimum creep rate and grain diameter for high purity copper tested at 626°C and 2.07 x 10^8 dynes/cm^2. Grain sizes produced by variable strain techniques. Specimens annealed at 600°C.

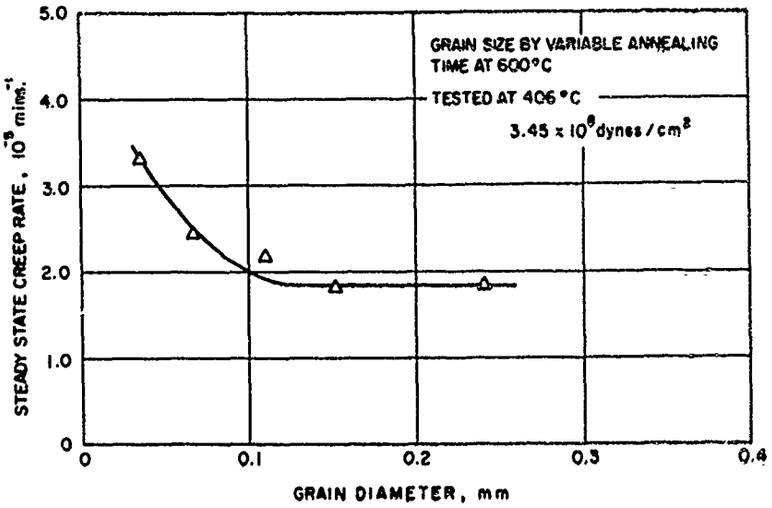

Fig. 7. The relationship between steady state creep rate and grain diameter for high purity copper tested at 406°C and 3.45 x 10^8 dynes/cm^2. Grain sizes produced by variable annealing time technique. Specimens annealed at 600°C.

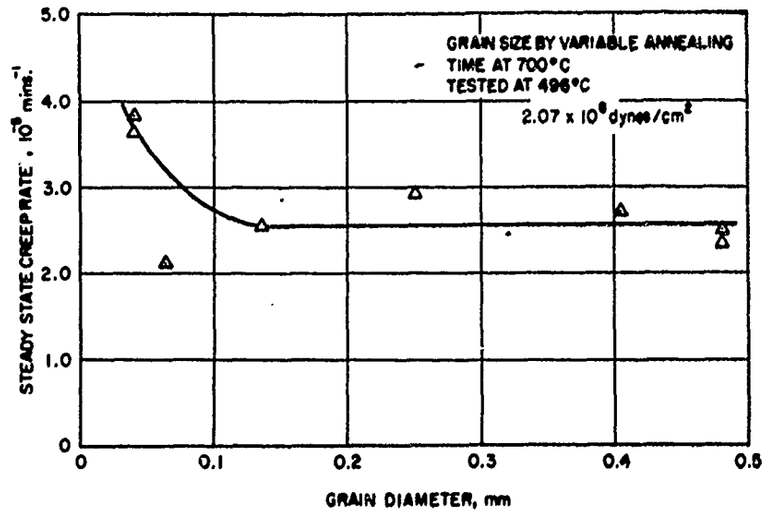

Fig. 8. The relationship between steady state creep rate and grain diameter for high purity copper tested at 495°C and 2.07 x 10^8 dynes/cm^2. Grain sizes produced by variable annealing time technique. Specimens annealed at 700°C.

three figures have two general characteristics in common: (1) a region where $\dot{\epsilon}_s$ decreases with increasing grain size and (2) a region where $\dot{\epsilon}_s$ is independent of grain size. Metallographic examination of specimens after testing showed that for samples deformed at 496°C and 626°C the grain sizes (Figures 5 and 6) in the region where $\dot{\epsilon}_s$ decreases with increasing grain size were unstable and experienced grain growth and/or recrystallization during testing. All other grain sizes were stable during testing including the grain sizes given in Figure 4 where $\dot{\epsilon}_s$ increases with decreasing grain size. The instability of the as-annealed grain size during testing was especially evident in the data shown in Figure 6. Creep curves for the fine grain sizes shown in this figure exhibited accelerated creep periods indicative of recrystallization during testing. Typical of these curves is the strain-time curve given in Figure 2 for the specimen designated by A in Figure 6. Creep curves for samples in Figure 5 which showed grain growth during testing gave no indication of any transient periods associated with recrystallization. As will be shown later, in Section A4, the small amount of grain growth that occurred during creep (about 20% increase for the finest grain size tested) probably had no effect on the macroscopically observed creep rate. On the other hand, the steep grain size dependence shown in Fig. 6 can probably be attributed to the influence of concurrent recrystallization on creep.

2. Variable Annealing Time Technique

The variation of $\dot{\epsilon}_s$ with grain size for grain sizes prepared by the variable annealing time technique is shown in Figures 7 and 8. The similarity between these data and the results given in Figures 4 and 5 is immediately obvious. The transition in creep behavior occurs at a

grain size approximately 0.1 mm in each set of data. The plateau region or regions where $\dot{\epsilon}_s$ is insensitive to grain size occurs at essentially the same creep rate in Figures 4 and 7 as well as in Figures 5 and 8 indicating no effect of annealing treatment on the creep rate - grain size relationship.

3. Variable Annealing Temperature Technique

Grain sizes prepared by annealing at various temperatures in hydrogen show an entirely different behavior than that observed in the previous figures. The data for specimens prepared by this method are given in Figure 9.

This peculiar behavior appears to be associated with the influence of hydrogen on the steady state creep rate. When copper samples were annealed in vacuum rather than in hydrogen a normal behavior was obtained as evident by the data presented in Figure 10. The steady state creep rates are lower for samples treated in a vacuum atmosphere than for samples treated in hydrogen. The magnitude of the creep rates obtained under vacuum annealing conditions correspond to the creep rates obtained by the variable strain technique (Fig. 5) and the variable time technique (Fig. 8). It is believed that the increasing solubility of hydrogen in copper with increasing temperature lead to the high steady state creep rates observed, although the exact mechanism of hydrogen weakening is not understood.

The unusual behavior of the hydrogen annealed samples could also be eliminated by aging such samples for a long time at low temperature. This was presumably due to a precipitation of hydrogen from solution or to its elimination by diffusion. Typical results from such an aging treatment are shown by the data given as filled circles in Figure 10. These data were obtained from copper samples initially annealed in hydrogen at

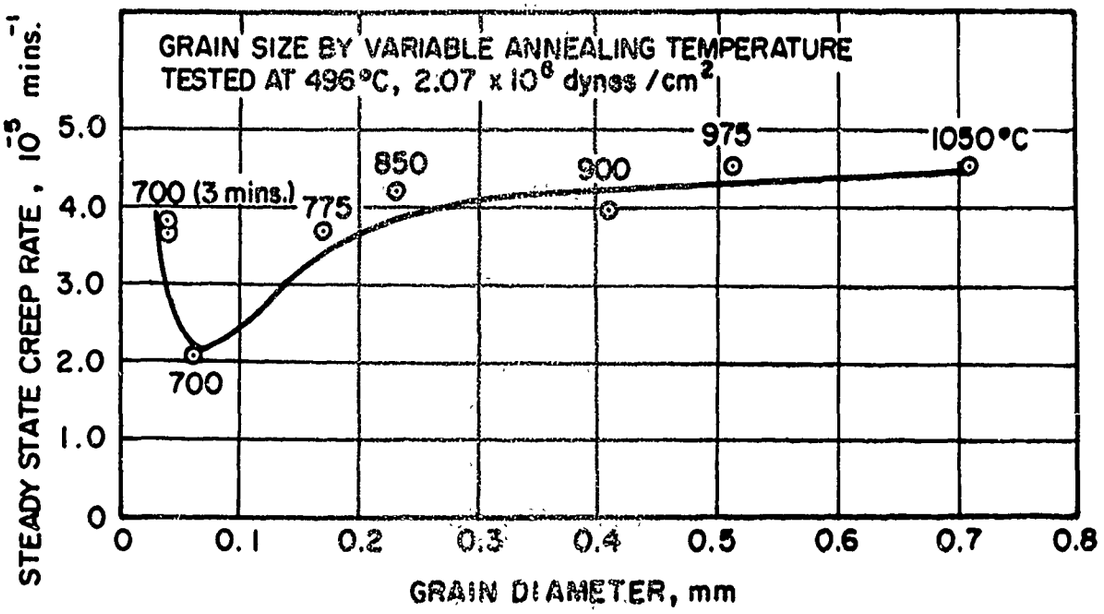

Fig. 9. The relationship between steady state creep rate and grain diameter for high purity copper tested at 496°C and 2.07 x 10^8 dynes/cm^2. Grain sizes produced by the variable annealing temperature technique. The temperatures listed indicate the specific annealing temperatures in hydrogen atmosphere (for 5 minutes except where indicated otherwise).

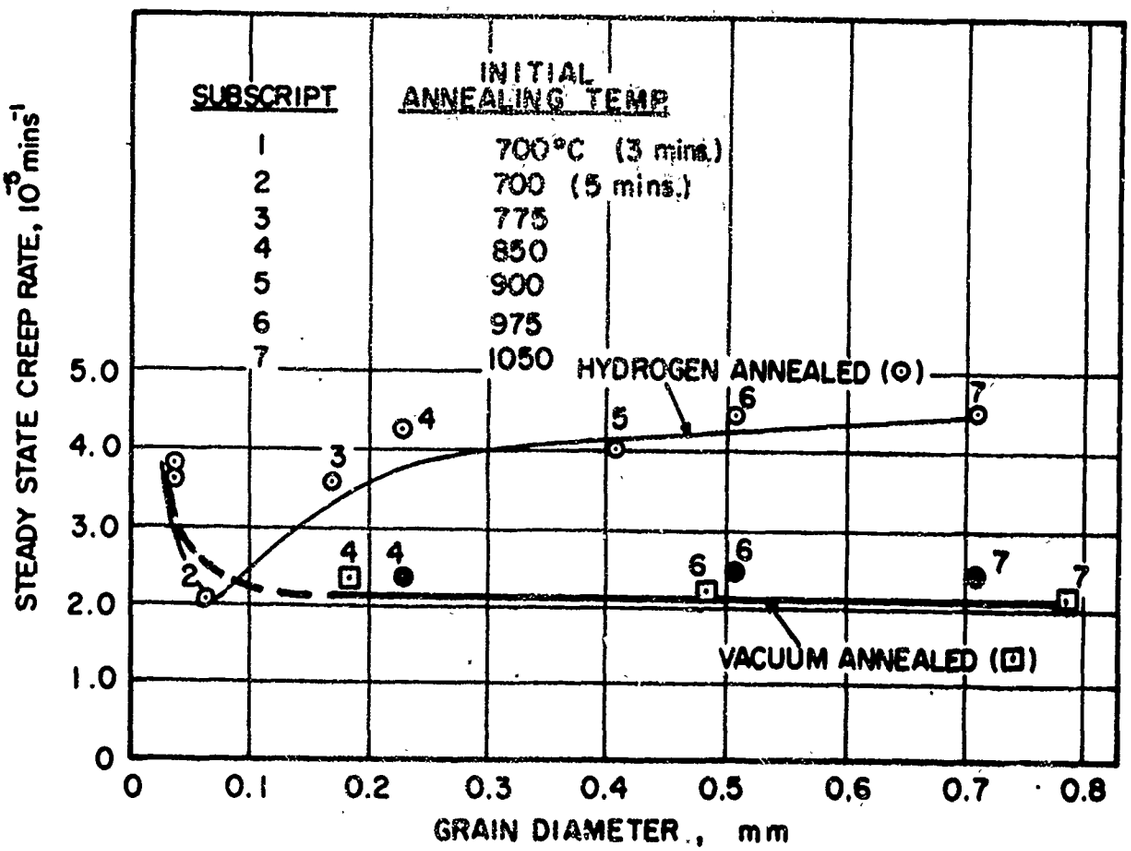

Fig. 10. Influence of aging treatments and annealing in vacuum on the relationship between steady state creep rate and grain diameter for grain sizes produced by the variable annealing temperature technique for high purity copper. Data given by filled circles represent samples which were annealed at the indicated temperature for five minutes in hydrogen and then further annealed at 700°C for 800 minutes.

various high temperatures and then subsequently annealed in hydrogen at 700°C for 800 minutes. The equilibrium hydrogen concentration is believed to be less than .002 atomic percent at 700°C[18]. The maximum effect this concentration of hydrogen has on the observed $\dot{\epsilon}_s$ can be taken as the difference in plateau regions of the vacuum annealed and the 700°C hydrogen annealed data given in Fig. 10. This difference is only about 15% and is within the range of experimental scatter of observed creep rates.

4. Low Angle Grain Boundary Samples

Data on the influence of grain boundary misorientation on $\dot{\epsilon}_s$ are presented in Figures 11 and 12. In these figures the steady state creep rates of both randomly oriented and [001] (100) textured (cube textured) copper of identical grain size are compared for stresses of 2.07×10^8 and 4.8×10^8 dynes/cm^2 in the temperature range of 400 to 542°C. The randomly oriented samples were prepared by cold rolling 50% and then annealing in vacuum for five minutes at 700°C. This is the same treatment used to obtain the finest grain size in Figure 5. The results indicate that the cube textured copper has a lower creep rate than the randomly oriented copper, regardless of the orientation of the tensile axis of the textured sample. The average difference in creep rates between cube textured and randomly oriented copper in Figures 11 and 12 is approximately a factor of two.

An interesting aspect of the data in Figure 11 concerns the problem of grain size instability during testing. The randomly oriented samples in this figure which were tested at or above 496°C exhibited some localized grain growth during testing. On the other hand, the [001] (100) textured specimens were stable at all temperatures during testing and there was no

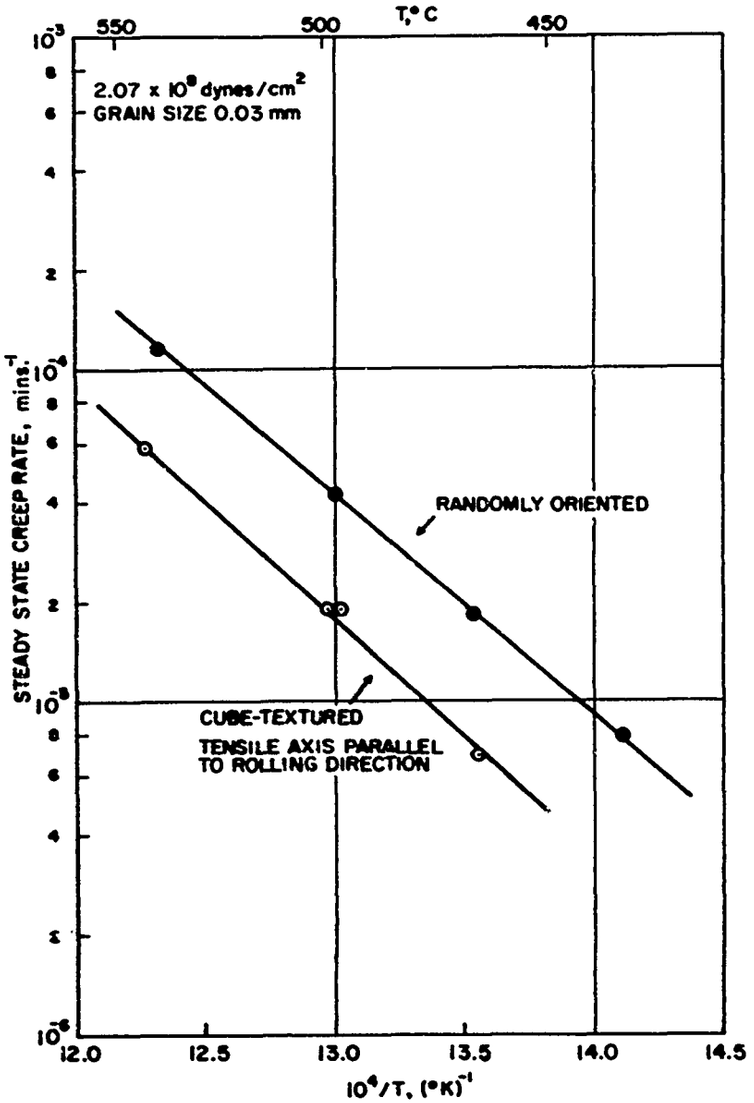

Fig. 11. The effect of temperature on the steady state creep rate of randomly oriented and cube textured high purity copper tested at 2.07 x 10^8 dynes/cm^2.

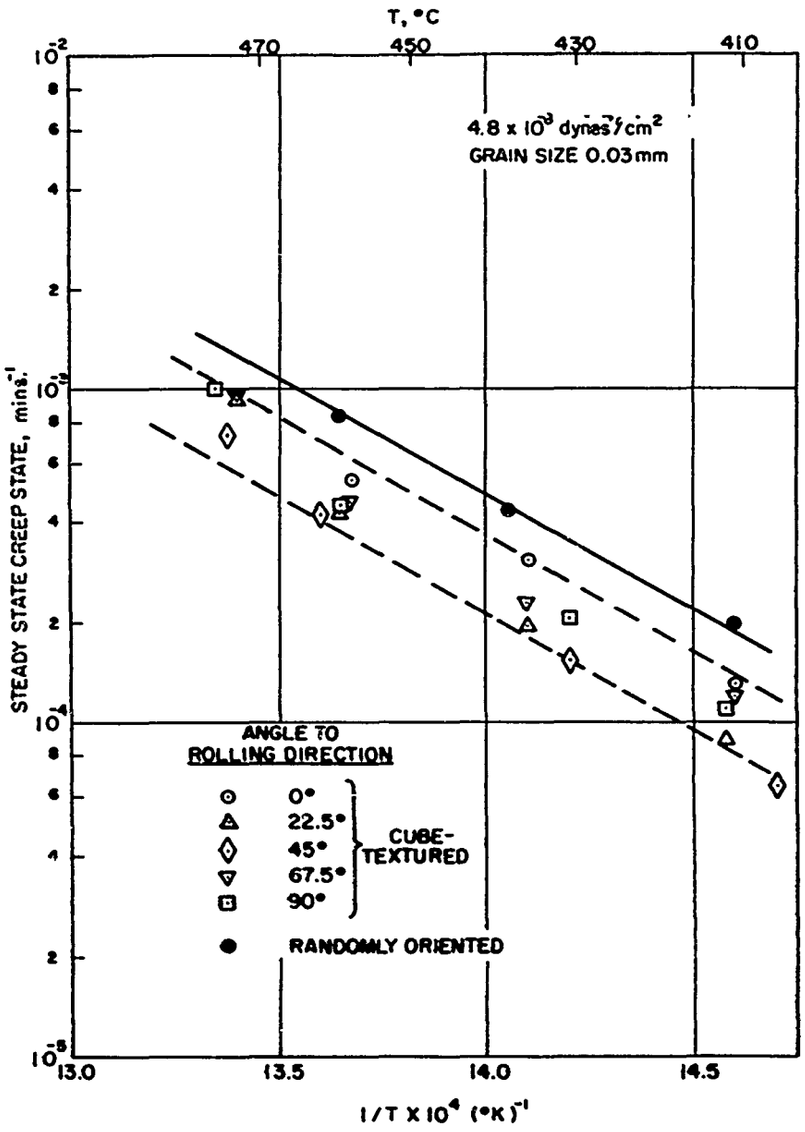

Fig. 12. The effect of temperature on the steady state creep rate of randomly oriented and textured high purity copper tested at 4.8 x 10^8 dynes/cm^2.

evidence of grain growth. If the localized grain size instability of the randomly oriented specimens resulted in an appreciable increase in the observed creep rate, then one would expect a different temperature relationship between the randomly oriented and textured samples in Figure 11 depending on whether or not grain growth occurred. The fact that the relationship between the two sets of samples is the same regardless of the fact that grain growth occurred for the randomly oriented samples tested at the two highest temperatures strongly suggests that this localized grain size instability has little or no effect on the macroscopically observed creep rate. Inasmuch as the randomly oriented grain size specimens tested to obtain the data in Figure 11 are identical with the finest grain size specimen reported in Figure 5, the above observation is an indication that the increasing creep rate with decreasing grain size observed in Figure 5 cannot be attributed to an increase in $\dot{\epsilon}_s$ due to the slight amount of grain growth that took place during testing.

B. GRAIN BOUNDARY SHEAR ANALYSIS

The grain size dependence of the contribution of grain boundary sliding to the total strain ϵ_{gb}/ϵ_t, was determined for three different grain sizes (prepared by the variable strain technique) at a stress of 2.07×10^8 dynes/cm^2 and temperature of 496°C. The three grain sizes studied were (1) a fine grain size (0.04 mm) which is in the region where $\dot{\epsilon}_s$ decreases with increasing grain size (see Figure 4), (2) an intermediate grain size (0.093 mm) which is in the transition area between the regions where $\dot{\epsilon}_s$ decreases with increasing grain size and where $\dot{\epsilon}_s$ is independent of grain size, and (3) a large grain size (0.22 mm) which is in the region where $\dot{\epsilon}_s$ is independent of grain size. Tests were run well into the steady state creep region in

order to determine the ratio ϵ_{gb}/ϵ_t during steady state creep. In addition, two tests were stopped at lower strains for the intermediate grain size to check the constancy of ϵ_{gb}/ϵ_t with strain. The results of these tests are summarized in Table 4.

It is apparent that the ratio ϵ_{gb}/ϵ_t behaves as a function of grain size almost in the same manner as $\dot{\epsilon}_s$. That is, ϵ_{gb}/ϵ_t is large for small grain sizes, then decreases with increasing grain size reaching an approximately constant value for large grain sizes. It is equally evident that for the range of strains investigated, the value of ϵ_{gb}/ϵ_t is independent of strain.

A cube textured sample was also tested at 496°C and 3000 psi and there was no evidence that any measurable grain boundary displacement occurred.

C. INFLUENCE OF GRAIN SIZE ON CREEP SUBSTRUCTURE

In this study only the dislocation substructure present during steady state creep was investigated. The grain sizes investigated were the same as those studied in the grain boundary shear analysis, namely 0.04, 0.093 and 0.22 mm. A cube textured sample with a <100> tensile axis was also investigated. Samples were examined by transmission electron microscopy after having been crept at 496°C and 2.07×10^8 dynes/cm^2. Observations were made primarily on the subgrain size and not on the dislocation density within subgrains.

The data concerning the influence of grain size on the subgrain diameter are listed in Table 5. The values listed are the average values determined from several different micrographs. The variation of individual determination from the average was about $\pm$ 25%. Thus, within experimental scatter the subgrain size is independent of grain size. The subgrain dia-

TABLE 4 - Experimental results of grain boundary shear analysis. Creep tests conducted at 496°C and 2.07×10^8 dynes/cm^2.

Grain Diameter mm	ϵ_t	ϵ_{gb}	ϵ_{gb}/ϵ_t
0.04	0.094	0.032	0.34
0.093	0.025	0.0047	0.19
0.093	0.052	0.0082	0.16
0.093	0.099	0.017	0.17
0.22	0.098	0.013	0.13

TABLE 5 - Subgrain size as a function of grain size for samples tested at 496°C and 2.07×10^8 dynes/cm^2.

Grain Diameter mm	ϵ	Subgrain Diameter, μ
0.03 [001](100)	0.138	3.3
0.04	0.094	3.1
0.093	0.061	3.5
0.22	0.098	3.4

meter of the cube textured sample is also nearly identical with that of the randomly oriented samples. A typical micrograph of the creep substructure is shown in Figure 13.

DISCUSSION

Before discussing the probable reasons for the observed $\dot{\epsilon}_s$ - grain size relationship it is convenient to compare the data of this investigation with the data in the literature. Two other investigators have studied the influence of grain size on creep rates in copper and both sets of data appear to disagree with the results of this investigation. Feltham and Meakin[13] have found that $\dot{\epsilon}_s$ increases with increasing grain diameter in the extremely narrow grain size range 0.02 to 0.04 mm. The data of Parker[15] indicate that $\dot{\epsilon}_s$ increases with increasing grain diameter for randomly oriented polycrystalline copper and decreases with increasing grain diameter for non-random (slightly textured) polycrystalline copper. The grain size range investigated by Parker was 0.025 to 0.14 mm.

The grain size study of Feltham and Meakin was conducted at 500°C and 4.9×10^8 dynes/cm^2. Their grain sizes were prepared by combinations of annealing times and temperatures from within the ranges 18-36 minutes and 550-700°C. On the basis of the present results it seems certain that some, and possibly all, of Feltham and Meakin's samples underwent extensive grain growth during testing, especially considering that some of their samples were annealed at only 50°C above the test temperature. This possibility was not considered by the authors and they apparently did not examine their specimens after testing to determine whether grain growth occurred. In fact, the data reported by Feltham and Meakin for two samples annealed at 700°C, which may have been the most stable grain sizes of the entire series, do show $\dot{\epsilon}_s$ to decrease with increasing grain size, in accord with the present observations.

Fig. 13. Typical creep substructure for high purity copper tested at 496°C and 2.07 x 10^8 dynes/cm^2. Grain diameter = 0.093 mm. ϵ = 0.061.

Feltham and Meakin also reported that there was a critical stress, the magnitude of which is temperature dependent, below which no grain size dependence of the steady state creep rate was observed. The data presented in the present study at 406 and 496°C are below this critical stress while the data at 626°C are above. As the behavior in all cases is similar, it is apparent that the present results do not support the existence of a critical stress.

There is no apparent way in which the data of Parker can be reconciled with the results of this study. The difference in results between the two investigations may, in part, be attributed to a difference in material purity of the copper used or to the difference in experimental setup. Parker used a helium atmosphere whereas the creep tests performed in this investigation were done in a dry deoxidized hydrogen atmosphere. Approximately the same stress and temperature conditions were used in both investigations.

To explain the general $\dot{\epsilon}_s$ - grain size relationship illustrated in Figure 3 it is necessary to understand why $\dot{\epsilon}_s$ is independent of grain size at large grain sizes and why it increases with decreasing grain size for small grain sizes. The magnitude of the effect we are trying to explain is small, only about a factor of two or three for the grain size range investigated, but it is a real and reproducible effect.

The possible roles grain boundaries may play in high temperature plastic deformation may be grouped under three general headings: (1) they may act as barriers to dislocation motion, (2) they may act as dislocation or vacancy sources and (3) they may contribute to the overall strain by grain boundary sliding (or localized deformation near the boundary). Each of these possibilities will be considered separately.

Grain boundaries as barriers. It is well established that not only grain boundaries but subgrain boundaries may act as barriers to dislocation motion. It is equally well established (not only in this investigation but also in several others[17, 19-21]) that the subgrain size formed during creep is independent of grain size. This means that the total grain boundary and subgrain boundary area per unit volume is independent of grain size. For the grain size range investigated it also means that the vast majority of this boundary area belongs to the subgrains, as the smallest grain size investigated was some ten times larger than the subgrain diameter observed at 496°C and 2.07 x 10^8 dynes/cm^2. Hence, regardless of the grain size, as long as it is reasonably large compared to the subgrain size, the dislocations are opposed by the same number of boundaries per unit volume. Also, the grain boundaries comprise such a small fraction of the total boundaries that even if the dislocations behave differently in the presence of high angle boundaries, it is unlikely that they would have a measurable effect on the observed creep rate. This concept predicts that $\dot{\epsilon}_s$ will be independent of grain size and discounts the postulate that grain boundaries are major barriers to dislocations. This latter argument predicts that $\dot{\epsilon}_s$ should be proportional to the square of the grain diameter.

Grain boundaries as dislocation or vacancy sources. The possibility that the grain size influences the mobile dislocation density has been proposed by Garofalo et al[11]. Assuming that during creep both grain boundaries and subboundaries act as sources and sinks of dislocations, they derive an expression for $\dot{\epsilon}_s$ at constant stress and temperature of the form

$$\dot{\epsilon}_s = \frac{k_1}{d} + k_2 d^2 \qquad (1)$$

where k_1 and k_2 are constants and d is the average grain diameter. The

validity of equation (1) requires that the mobile dislocation density vary with grain size. To date there is no experimental confirmation of this behavior. In fact, measurements by Barrett, Nix and Sherby[22] on an Fe-3.0% Si alloy, where a range of grain sizes from 0.05 to 0.3 mm was studied, showed the dislocation density within subgrains to be independent of grain size. The validity of equation (1) is therefore seriously questioned.

The possible influence of grain boundaries as sources of vacancies on the creep resistance of materials has been considered by Parker[15]. High angle boundaries are known to be good sources of vacancies whereas low angle dislocation boundaries are believed to be much less effective[23]. If dislocation climb is dependent on the ready availability of vacancies from boundary sources, then the higher creep rate of non-textured polycrystalline copper over textured polycrystalline copper (Fig. 11 and 12) can be explained by this mechanism. However, such a mechanism cannot explain the insensitivity of the steady state creep rate to the grain size in the grain size range 0.1 to 0.7 mm, nor can the Parker mechanism explain the variation of creep rate with grain size for grain sizes below 0.1 mm when grain sizes are prepared by the variable strain technique (Figs. 4-6).

Grain boundary shearing. The discussion presented above indicates that if creep takes place predominantly by the motion of dislocations, then the steady state creep rate should not depend on the grain size. If the process of grain boundary shear is important in determining the overall extension rate, however, $\dot{\epsilon}_s$ will probably be a function of grain size in that the grain boundary area per unit volume increases as the grain diameter decreases. There are several reasons to suspect that the increase in $\dot{\epsilon}_s$ with decreasing grain size observed in this study is due to the in-

creasing importance of grain boundary shearing as a deformation mechanism at small grain sizes.

The contribution of grain boundary sliding to the total strain was observed to be a strong function of grain size (Table 5). In fact, the influence of grain size on ϵ_{gb}/ϵ_t was similar to its influence on $\dot{\epsilon}_s$. For small grain sizes ϵ_{gb} comprised 34% of the overall elongation.* Assuming that the rate of strain due to the motion of dislocations is the same in all samples, independent of grain size, this increased component of ϵ_{gb} for the fine grain sizes should give rise to an overall increase in $\dot{\epsilon}_s$. Further evidence in support of this concept is as follows. The fine grained, cube textured specimens, for which ϵ_{gb} was negligible, crept at rates comparable to those of the large grain size randomly oriented samples, for which the magnitude of ϵ_{gb} was also small. In addition, the relative difference between the creep rates of the cube textured material with a <100> tensile axis and the randomly oriented material in Figures 11 and 12 is observed to decrease with increasing stress. This is in accord with the numerous observations[24-28] that the contribution of ϵ_{gb} to the total strain decreases as the stress is increased.

The above observations are significant in that they indicate grain size is not an important factor in determining the observed creep rate of pure metals. It is likely that the results of earlier studies which have shown $\dot{\epsilon}_s$ to be a strong function of grain size are somewhat misleading for two reasons. First, the materials investigated were not

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* The values of ϵ_{gb}/ϵ_t may not be exact values in that it has not been established that surface observations of ϵ_{gb} agree with internal marker measurements over the grain size range covered in this investigation. However, the data in Table 5 is evidence that grain boundary shearing is more important as a deformation mechanism as the grain size is reduced.

pure metals and the treatments used to obtain different grain sizes probably resulted in different impurity distributions. It is also possible that the small grain sizes investigated were unstable during testing and experienced grain growth and/or recrystallization. As illustrated in Figure 6, a grain size instability can give rise to an exaggerated grain size effect.

SUMMARY

The effect of grain size, over a range in grain diameters between 0.03 and 0.7 mm, and annealing treatment on the steady state creep rate of high purity copper has been studied at 406, 496 and 626°C under constant stress conditions. The observations led to the following conclusions:

(1) The subgrain diameter for copper tested at 2.07×10^8 dynes/cm^2 and 496°C is independent of grain size and equal to approximately 3.3 microns.

(2) The contribution of grain boundary shearing to the total strain, ϵ_{gb}/ϵ_t, increased with decreasing grain size. For samples tested at 2.07×10^8 dynes/cm^2 and 496°C, ϵ_{gb}/ϵ_t was equal to 0.34 for a grain diameter of 0.04 mm and equal to about 0.15 for grain diameters of 0.1 mm or larger. Polycrystalline cube textured copper (grain diameter = 0.03 mm) did not experience measurable grain boundary shear. The ratio ϵ_{gb}/ϵ_t was independent of strain.

(3) The relationship between the steady state creep rate and grain size was found to be the same, independent of the technique used to prepare the various grain sizes. For specimens with grain diameters above about 0.1 mm, $\dot{\epsilon}_s$ appears independent of grain size, whereas for small grain diameters, $\dot{\epsilon}_s$ increases slightly with decreasing grain size. The slight in-

crease in $\dot{\epsilon}_s$ with decreasing grain size is attributed to the increasing importance of grain boundary sliding as a deformation mechanism as the grain size is decreased. The relative constancy of $\dot{\epsilon}_s$ with grain size, for grain diameters above 0.1 mm, is explained by the presence of the same fine subgrain structure developed during primary creep and to the same fractional contribution of grain boundary shearing to creep independent of grain size. Polycrystalline cube textured copper, which contains low angle grain boundaries, exhibits steady state creep rates that are lower than those observed in randomly oriented copper of the same grain size. This observation can be explained by negligible contribution of grain boundary shearing to creep in the textured polycrystalline material.

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